S/N 09/614,631

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	HILGREN ET AL.	Examiner:	J. PAK
Serial No.:	09/614,631	Group Art Unit:	1653
Filed:	JULY 12, 2000	Docket No.:	163.1382US01
Title:	METHOD AND COMPOSITION FOR INHIBITION OF MICROBIAL GROWTH IN AQUEOUS FOOD TRANSPORT AND PROCESS STREAMS		

CERTIFICATE UNDER 37 CFR 1.6(d):
I hereby certify that this paper is being transmitted by facsimile to the U.S. Patent and Trademark Office on January 27, 2006.

By: Kay Fahland
Name: Kay Fahland

DECLARATION UNDER 37 CFR § 1.131

Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

I, John D. Hilgren, declare and state the following:

1. I am an inventor of the subject matter of the patent application identified above and an employee of Ecolab, Inc., the assignee of the patent application identified above.

2. I understand that the Examiner has cited Hei (US 6,024,986) as prior art in prosecution of the application identified above. I understand that the Hei patent was filed on May 24, 2999.

3. I further understand that the filing date of the present patent application Serial No. 09/614,631 is July 12, 2000.

4. I state that before the date of the Hei patent, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above.

5. The claims of the patent application identified above relate to compositions including:

about 35 to about 45 weight-% acetic acid, about 5 to about 15 weight-% octanoic acid, about 3 to about 8 weight-% hydrogen peroxide, about 8 to about

16 weight-% peroxyacetic acid, about 1 to about 5 weight-% peroxyoctanoic acid, and about 0.1 to about 2 weight-% chelating agent;

about 40 weight-% acetic acid, about 10 weight-% octanoic acid, about 5 weight-% hydrogen peroxide, about 12 weight-% peroxyacetic acid, about 3 weight-% peroxyoctanoic acid, and about 0.6 weight-% chelating agent;

about 10 to about 150 ppm acetic acid, about 5 to about 40 ppm octanoic acid, about 4 to about 20 ppm hydrogen peroxide, about 5 to about 50 ppm peroxyacetic acid, about 2 to about 25 ppm peroxyoctanoic acid, and about 0.2 to about 2.5 ppm chelating agent;

about 133 ppm acetic acid, about 33 ppm octanoic acid, about 17 ppm hydrogen peroxide, about 40 ppm peroxyacetic acid, about 10 ppm peroxyoctanoic acid, and about 2 ppm chelating agent;

about 50 to about 60 weight-% acetic acid, about 10 to about 20 weight-% octanoic acid, about 5 to about 15 weight-% hydrogen peroxide, and about 0.3 to about 1 weight-% chelating agent; or

about 54 weight-% acetic acid, about 14 weight-% octanoic acid, about 10 weight-% hydrogen peroxide, and about 0.6 weight-% chelating agent;

Each claimed composition also has at least about 1 part by weight of peroxyoctanoic acid for each about 5 parts of peroxyacetic acid.

The present patent application includes at page 15 several tables describing embodiments of the claimed compositions. Two of the tables describe concentrate and use compositions including:

Chemical	Wt-%	ppm
Acetic Acid	40	133
Hydrogen Peroxide	5	17
HEDP	0.6	2
Octanoic Acid	10	33
Peroxyacetic Acid	12	40
Peroxyoctanoic Acid	3	10

One of the tables describes raw materials that can be used to make the concentrate composition described above. These raw materials include:

Raw Material	Weight %
Glacial Acetic Acid	54
Hydrogen Peroxide, 35%	30
HEDP, 60%	1
Octanoic Acid, 95%	15

6. During the research and development leading to the claimed compositions, and before May 24, 1999, my coinventors and I made several compositions meeting the limitations of the claims of the present application and including the compositions described in the tables in paragraph 5 above.

7. Accompanying Exhibit A reports studies of compositions that meet the limitations of the present claims and including the compositions described in the Tables in paragraph 5 above. In Exhibit A, the initial, concentrate, and use compositions described in the tables in paragraph 5 above are referred to as "Falcon 15 O". The composition called "Falcon 15 AE" also meets the limitations of the present claims. The contents of these formulas can be found at page 9-10 of Exhibit A. Page 9 of Exhibit A describes the stability of these formulas. The pages of the report attached as Exhibit A were prepared before May 24, 1999.

8. The evidence presented in Exhibit A indicates that, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above. Moreover, Exhibit A demonstrates the concentrations of the compositions called Falcon 15 O and Falcon 15 AE, as invented prior to May 24, 1999, fit within the claims of the present invention. Pages 1 and 9 of exhibit A also demonstrates the compositions of Falcon 15 invented prior to May 24, 1999, were physically stable.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

John D. Hilgren

1-25-06

EXHIBIT A
DATED DECEMBER 23, 2005
APPLICATION NO. 09/614,631

TITLE F-15 THALCON 15 PROJECT NO.

W-299 BOOK NO. 9980 34

That does not require a coupler NAS

Work continued from Page

OBJECTIVE: Develop a concentrated version of Falcon KX 6049. Or develop a sanitizer concentrate that maximizes actives (POAA, POOA, H_2O_2) while minimizing costs.

Discussion w/ [redacted]: [redacted] has developed a "Falcon 15" formula that is physically stable. [redacted] Formula A)

	AM	# Equilibrium
Glacial Acetic Acid	59.0%	50.87
H_2O_2 (35%)	25.0	3.50
Dequest 2010 (60% Active)	1.0	0.60
Octanoic Acid	15.0	12.28
~~POAA~~ POAA	0.0	10.30
POOA	0.0	3.02
Water	0.0	19.07

The Equilibrium concentrations were based on the "SOLVER" computer program. We chose equilibrium constants based on @ 3 samples of Falcon 15 produced and analyzed by [redacted]. $K_1 = 1.7$ & $K_2 = 2.35$

FALCON 16 EQUILIBRIUM CONSTANTS DERIVATION

Using these Keq's.

RICHTER/REINHARDT FORMULA

	% Raw Mat'l
Acetic Acid	59.00
Hydrogen Peroxide	8.75
Dequest 2010 (60%)	0.60
Octanoic Acid	15.00
POAA	
POOA	
Water	16.65
	100.00

[redacted] 1.0443

Concentration in Molarity

	9807-48	9889-36	9889-48
AAi	10.27	10.27	10.27
H_2O_2i	2.69	2.69	2.69
OAi	1.09	1.09	1.09
H_2Oi	9.66	9.66	9.66
AAeq	8.98	8.04	7.78
H_2O_2eq	1.02	0.99	1.01
OAeq	0.87	0.78	0.76
POAAeq	1.34	1.43	1.38
POOAeq	0.30	0.20	0.19
H_2Oeq	10.56	14.88	16.16
K1 =	1.54	2.69	2.65
K2 =	3.54	3.95	4.05
K3 (from eq)	2.30	1.47	1.42

ANALYTICAL RESULTS

**After Manipulation of SOLVER Program we decided: K1 = 1.7 & K2 = 2.35

K1=2.77 K2=4.00 calculated based on Analytical

2 Week Equilibrium Concentrations (% By Weight)

9807-48	9889-36	9889-48	SOLVER	SOLVER
51.57	46.19	44.70	50.87	49.54
3.33	3.21	3.29	3.50	2.62
0.60	0.60	0.60	0.60	0.6
12.00	10.80	10.50	12.28	11.76
9.73	10.41	10.10	10.30	11.98
4.57	3.14	2.85	3.02	3.6
16.20	25.65	27.86	19.57	20.13
100.00	100.00	100.00	100.24	100.29

$AA + H_2O_2 \xrightarrow{K1} POAA + H_2O$

$OA + H_2O_2 \xrightarrow{K2} POOA + H_2O$

$POAA + OA \xrightarrow{K3} POOA + AA$

K1 ave of last two: 2.77
K2 ave of last two: 4.00
K3 ave of last two: 1.44

SIGNATURE [redacted]

DISCLOSED TO AND UNDERSTOOD BY [redacted] DATE [redacted] WITNESS DATE 1a-27-06

TITLE Falcon 15

PROJECT NO.

BOOK NO. 9980

Work continued from Page 34

According to [redacted], original goal was to maximize octanoic acid content in raw material but they determined a solubility limit near 15%. Physical stability tests are performed at 40°F and 100°F

It appears from Micro testing that the lower the H_2O_2 the better. We will focus on ① minimizing cost ② maintain Physical Stability ③ Maintain Chemical Stability ④ maximize PA/PAA ratios ⑤ Understand other factors involved that increases efficacy ⑥ Make Non-Detonable solutions.

I. Physical Stability TESTS

A) CONCENTRATE
① Room Temp.
② 40°F
③ 100°F - 1 MONTH
I. Qualitative - Turbidity
II. Quantitative - G.C

B) Use Solution - May use Analytical G.C. To determine if octanoic separates to the top.
① Room Temp
② 40°F
③ 100°F

II. Chemical Stability TESTS
- CONCENTRATE ONLY
① Room Temp.
② 100°F

Method of Analysis
① ATM 202
② HPLC.

III. EFFICACY
① - At use concentrations
- At exposure times / Temp. / Etc.

Work continued to Page 36

SIGNATURE [redacted] DATE [redacted]

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TITLE [illegible] FALCON 15

PROJECT NO.

BOOK NO. 9980

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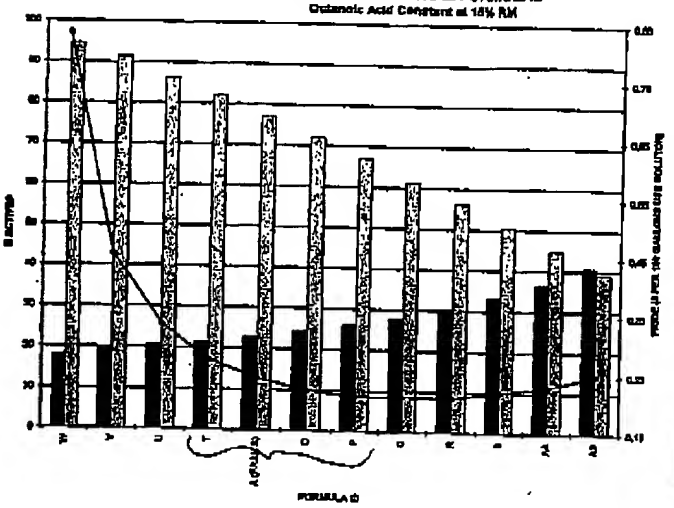
Work continued from Page 35

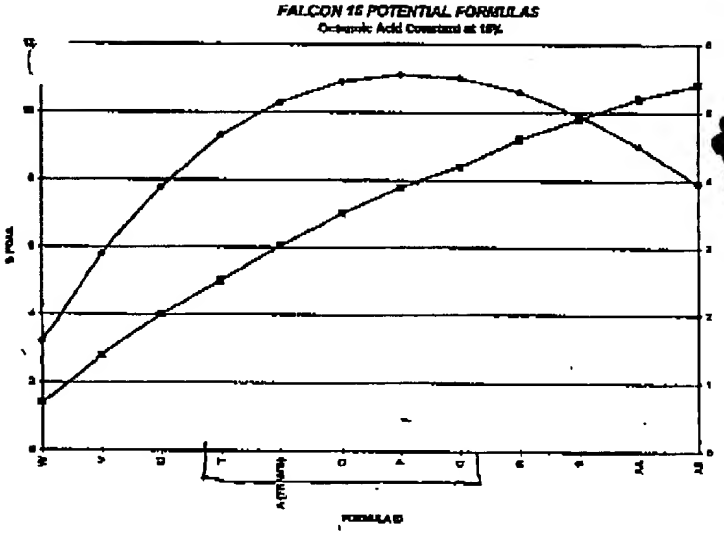
Important Areas of INTEREST WHEN DESIGNING NEW & FALCON 15 FORMULA:



	AA	H2O2
W	79%	5%
V	74%	10%
U	69%	15%
T	64%	20%
A(Prev)	59%	25%
O	54%	30%
P	49%	35%
Q	44%	40%
R	39%	45%
S	34%	50%
AA	29%	55%
AB	24%	60%

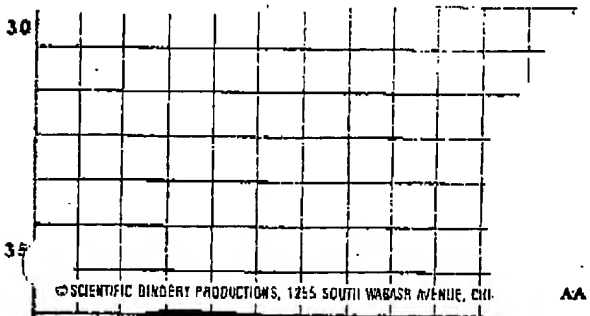
[redacted]

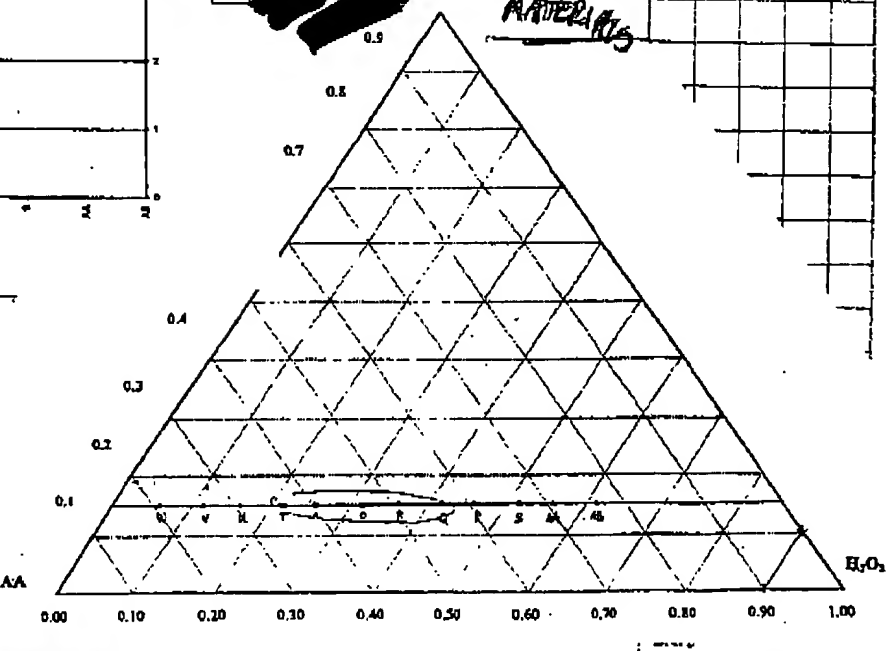
* For Ternary diagram, determine where the detonable formulas are and where the unstable (physically) formulas are.



[redacted]

RAW MATERIALS





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DATE [redacted]

WITNESS

DATE [redacted]

TITLE Falcon 15 PROJECT NO. 37

BOOK NO. 9980

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Physical Stability TESTING: MIX ALL SAMPLES WITH Tap H2O FOR 5 minutes - Stir Plate

CONCENTRATION	TEMP.	QUALITATIVE ANALYSIS
FALCON 15 - CONCENTRATE Prepared by Joel Schilling 9/19/96 SG = 1.0443	100°F	START DATE: 12-27-96
1oz/13 gallons 601 ppm (v/v) Falc = 0.5815 gms 628 (w/w) H_2O = 925.8 gms	~~Room Temp~~ 60°F	MIX w/ Tap H_2O / A small amount of oil drops seemed to be on the surface, but after mixing not as evident
1202 ppm (v/v) Falc = 1.0175 1255 ppm (w/w) H_2O = 810.2	~~Room Temp~~	Once mixed appears Stable
1803 (v/v) Falc = 1.8075 1883 (w/w) H_2O = 958.4	60°F ~~Room Temp~~	An obvious oil separation on top before thoroughly mixing. After mixing very slight oil droplets.
2253 (v/v) Falcon = 1.9030 2331 (w/w) H_2O = 815.2	60°F ~~Room Temp~~	Oil separation on top before mixing. Obvious oil slick after mixing
6695 (v/v) Falcon = 5.610 6992 (w/w) H_2O = 798.2	60°F ~~Room Temp~~	Heavy oil slick prior to mixing and after mixing
9576 10% (w/w) Falcon = 8.1305 H_2O = 805.1	60°F ~~Room Temp~~	Heavy oil slick before and after mixing

(SEE pp. 40)

* Appears that solubility limit for this Falcon 15 ([redacted]) formula is ~~between~~ around 1800 ppm (v/v) at 60°F.

* Could we use a H_2O soluble dye to stand out greater?

$$\frac{10^6}{(X\text{ ppm})(128)} = X \text{ gallons for 1 oz}$$

Work continued to Page

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PROJECT NO. 38

CHEMICAL STABILITY BOOK NO. 9980

Work continued from Page

Objective: Determine if chemical Stability changes substantially over time at elevated temperatures.

SOLUTION: [redacted] Falcon 15 prepared [redacted]

Analytical Results from [redacted]: OA - 8.73%, POOA - 2.67%, H_2O_2 - 4.12%, POOA - 12.30% } LC

Formula
59% - HOAc
25% - H_2O_2 (35%) - 8.75% (100%)
15% - OA
1% - Dequest 2010

Analytical Results

INITIAL COMPONENTS	[redacted]	Room TEMP. Start of Test ([redacted])	Room TEMP End of 1 month (~~Equivalent to~~ over 30 days)	100°F End of 1 MONTH (Removed from oven [redacted])
Acetic Acid	N/A	~~59.80~~ 50.67	51.22	63.0%
H_2O_2	4.12%	~~3.80~~ 3.20	1.50	<0.1%
POOA	~~8.39%~~ 11.03%	~~10.20~~ 9.13	7.88	0.40%
POOA	2.67%	~~2.26~~ 2.26	2.24	0.27%
OA	8.73%	~~12.20~~ 12.20	13.0	18.17%
		~~0.6% Dequest~~ 0.6% Dq. 11.74% H_2O 21.94% H_2O		

OVEN START DATE: [redacted]
END DATE: [redacted]

Work continued to Page

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TITLE

PROJECT NO.

39

Determine Upper OA limit

BOOK NO. 9980

Work continued from Page

Objective: Prepare the following Falcon 15 formulas to determine physical - stability of high OA concentrations.

FORMULA	Room Temp Stability	40°F Stability (If Room Temp is stable)
X - 57% - AA - 41.09gms 20% - H_2O_2 - 13.95 20% - OA - 13.90 1% - Dequest C - 0.74	STABLE CLEAR SOLUTION	STABLE CLEAR SOLUTION
Y - 49% - AA - 34.53gms 30% - H_2O_2 - 21.18 20% - OA - 14.10 1% - Dequest - 0.71	Using 40°F H_2O_2 - When mixing was not stable, but once at room temp. became stable & clear.	NOT STABLE Separated into two layers.
Z - 39% - AA - ~~34.53~~ 27.37 40% - H_2O_2 - ~~28.24~~ 28.24 20% - OA - ~~14.10~~ 13.68 1% - Dequest ~~0.71~~ 0.71	NOT STABLE - Top Octanoic Acid Layer.	Did not test - No need to.

*Depending on stability or instability of these formulas we will vary OA concentration.

Glacial Acetic Acid - 124016 LOT #115371
35% H_2O_2 - 240317 LOT B5762-144
Dequest ADPA - GOAW 250282 LOT# 05082-05802

Octanoic Acid - 190280 LOT 119455

Work continued to Page 40

SIGNATURE [redacted]

DISCLOSED TO AND UNDERSTOOD BY [redacted]

DATE [redacted]

WITNESS

DATE [redacted]

DATE

Determine Upper OA Limit.

Work continued from Page 37

According to "Merck Index" Octanoic Acid has a maximum solubility of 0.068 gms / 100 gms H_2O or (680 ppm w/w). It has greater solubility in acetic acid.

680 ppm (w/w) octanoic acid would be equivalent to

$$\frac{680}{(1.0943)(.1228)} = 5,303 \text{ ppm (v/v) Falcon 15 @ } 25^\circ C \text{ or Room Temperature.}$$

(1.0943 ↑ S.G.; .1228 ↑ % Octanoic Acid by "Solver")

* We noticed solubility problems at 1800 ppm (v/v) although this was at 16°C or 60°F.

* At room temp. we would expect to see slightly better solubility than 5303 due to the coupling ability of acetic acid. Although we are not sure of the solubility limit of peroctanoic acids. - Most likely no limits.

* The samples from pp: 37 were also placed in 40°F refrigerator

All samples use [redacted] Falc. 15 [redacted]

	STABILITY 40°F		STABILITY 60°F		STABILITY 70°F	
601 ppm (v/v)	STABLE	Sample of Solution Prepared	STABLE	Actual Raw Material - Whole Samples	STABLE	Sample of Solution Prepared
1202	STABLE		STABLE		STABLE	
1803	STABLE		UNSTABLE		STABLE	
2232	STABLE		UNSTABLE		STABLE	
6695	UNSTABLE		UNSTABLE		UNSTABLE	
9576	UNSTABLE		UNSTABLE		UNSTABLE	

*NOTE: The 40°F stability tests were performed by sampling from a larger sample while mixing. The solution may have already separated. Repeat experiment with large sample

Work continued to Page 41

SIGNATURE [redacted] DATE [redacted]

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Work continued from Page 40

Additional Concentrates to narrow in on Formulas - Determining Physical Stability.

FORMULA	Room Temp. STABILITY	40°F STABILITY
Q AA - 44% - 56.79 gms H_2O_2 - 40% - 51.64 Dequest - 1% - 1.29 DA - 15% - 19.47	NOT STABLE	NOT STABLE
R AA - 39% H_2O_2 - 45% Dequest - 1% DA - 15%	ASSUME INSTABILITY BASED UPON Q & the fact that AA is a Coupler	
Y1 AA - 52% - 54.58 gms H_2O_2 - 30% - 31.50 Dequest - 1% - 1.15 DA - 17% - 17.86	STABLE	[redacted] STABLE
Y2 AA - 49% H_2O_2 - 33 Dequest - 1% DA - 17%	Assume Instability due to P & Y being Unstable - Did not Prepare this sample	
Z1 AA - 39% H_2O_2 - 43% Dequest - 1% DA - 17%	Assume INSTABILITY due to Q being UNSTABLE and the fact that AA is a Coupler	
Z2 AA - 42% H_2O_2 - 40% Dequest - 1% DA - 17%		

RAW MAT'L PRODUCT LOT #'S

* Glacial Acetic Acid: 1049016 Lot #115371 Octanoic Acid: 190280 Lot 2190135
35% H_2O_2: 240317 Lot B5702-144
Pigment AOPA: 250282 Lot 05086-0580DR

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TITLE CONTD P3 of 5 PROJECT NO. 45

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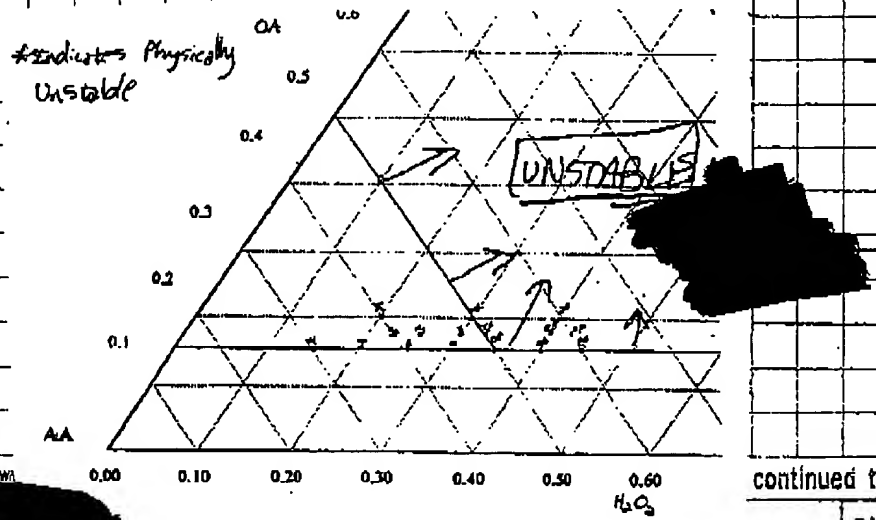
Formula	Room Temp STABILITY	40°F STABILITY
P AA – 49% – 51.65 H_2O_2 – 35% – 37.0 Dequest – 1% – 1.05 QA – 15% – 15.81	~~UNSTABLE~~ STABLE	UNSTABLE
X_2 AA – 57% – H_2O_2 – 23% – Dequest – 1% – QA – 17% –	Assumed STABILITY Based upon Stability of X_1 & X_3	
X_3 AA – 56% – 53.16 H_2O_2 – 24% – 24.72 Dequest – 1% – 1.00 QA – 17% – 16.24	STABLE	STABLE

*STABILITY CURVE THUS FAR:



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TITLE CONT'D FS-10

PROJECT NO. 46

BOOK NO. 9980

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* Based upon efficacy data from [redacted] - lower H_2O_2 proves to be more efficacious.

		Room Temp. STABILITY	40°F STABILITY
AC:	AA - 49.0 - 36.46 grms		
	H_2O_2 - 40.0 - 29.77	STABLE	UNSTABLE
	Dequest - 1.0 - 0.75		
	DA - 10.0 - 7.45		
AD:	AA - 39.0 -	Did Not Prepare based upon AC - This will be UNstable	
	H_2O_2 - 50.0 -		
	Dequest - 1.0		
	DA - 10.0		
AE:	AA - 57.0 - 46.09		
	H_2O_2 - 30.0 - 23.44	STABLE	STABLE
	Deq. - 1.00 - 0.82		
	DA - 10.0 - 7.81		
AE:	AA - 69.0 - 48.51		
	H_2O_2 - 20.0 - 14.03	STABLE	STABLE
	Deq. - 1.0 - 0.74		
	DA - 10.0 - 7.04		
A:	AA - 59.0 - 39.21		
	H_2O_2 - 25.0 - 16.63	STABLE	STABLE
	Deq. - 1.0 - 0.66		
	DA - 15.0 - 10.00		
O:	AA - 54.0 - 32.57		
	H_2O_2 - 30.0 - 18.11	STABLE	STABLE
	Deq. - 1.0 - 0.62		
	DA - 15.0 - 9.05		
T:	AA - 64.0 - 43.31		
	H_2O_2 - 20.0 - 13.54	STABLE	STABLE
	Deq. - 1.0 - 0.69		
	DA - 15.0 - 10.16		

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TITLE CONT'D

47

BOOK NO. 9980

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FORMULAS SENT TO ANALYTICAL FOR ANALYSIS:

FORMULA ID	DATE Prepared	[redacted]	ANALYTICAL		RESULTS	DATE:
		OA Acetic Acid	Acetic Acid	H_2O_2	POAA	POOA
"AF" AA - 69% H_2O_2 - 20% OA - 10%	[redacted]	8.58	58.48	1.79	10.92	1.22
"T" AA - 64% H_2O_2 - 20% OA - 15%	[redacted]	11.2	63.28	1.89	10.92	1.65
"X" AA - 59% H_2O_2 - 20% OA - 20%	[redacted]	15.8	47.49	1.92	10.11	2.72
" " AA - 59% A H_2O_2 - 25% OA - 15%	[redacted]	12.2	48.14	2.98	10.16	2.19
" AA - 57% AE H_2O_2 - 30% OA - 10%	[redacted]	7.45	46.64	3.98	13.46	1.76
" AA - 54% O H_2O_2 - 30% OA - 15%	[redacted]	11.9	40.99	4.15	13.11	2.51

NOTE: AF, T, A, AE & O - Had Equilibration @ room temp for 3day
X - " " " " " " 27day

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